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Some Reactions of Dialkylaminoborons

by

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Consideration of the established hydroboration, chloroboration and phenylboration reactions prompted attempts to extend this concept to aminoboration. Although addition to ethylenic bonds did not occur, it was found that addition to organic isocyanates and isothiocyanates readily occurred to give the expected boroureas. When applied to boron isocyanates and isothiocyanates however, disproportionations involving the boron substituents prevailed in good yield. Ultimately by such disproportions examples of the novel $\phi B(NR_2)NCX$ series of compounds were prepared.

Three types of reactions involving the addition of borine and its derivatives to carbon-carbon unsaturation had been described when this research was undertaken. These included the well known hydroboration reaction and the recently reported instances of chloroboration and phenylboration (3). Intrigued by the possibility that the scope of these reactions could be broadened to include aminoboration, the reactions of tris(dimethylamino)boron and bis(dimethylamino)phenylboron with several olefins were attempted. Like boron trichloride and phenylboron dichloride, these aminoborons induced the polymerization of cyclohexene but unlike the chlorides, did not add to nor-bornadiene.

Concurrent with the above, the reaction of these aminoborons with phenylisocyanate was attempted since here a 1,2 addition could also be expected. In these instances exothermic reactions occurred to give the expected boroureas in good yield according to the following reaction.

$$3\ C_6H_5NCX + B\left[N(CH_3)_2\right]_3 \longrightarrow B\left[C_6H_5NCXN(CH_3)_2\right]_3 \qquad 1.$$

I

a. - X = O

b. - X = S

These reactions were extended to include the use of phenylisothiocyanate to give the corresponding borothioureas. It was also found that the similar tetrakis(dimethylamino)diboron would readily add four moles of phenylisocyanate or phenylisothiocyanate.

To prove that the addition proceeded as shown in the reaction above and not in the reverse manner, the tris-borourea (Ia) was subjected to ethanolysis which readily occurred giving triethylborate and phenyldimethylurea in quantitative yield. The corresponding thiourea was more resistant to alcoholysis and required refluxing in aqueous dioxane to give phenyldimethylthiourea and benzene boronic acid.

At this time, Beyer, Niedenzu and Dawson reported (1) identical observations in the cases of bis(dimethylamino) phenylboron and tetrakis(dimethylamino)diboron and also established the stepwise reaction of the aminoboron groups. Also Lappert advised the authors of his successful application of the chloroboration and phenylboration reactions in the isocyanate system (4). Consequently effort in this area was stopped although it should be reported that the reaction of bis(dimethylamino) phenylboron with toluene diisocyanate or hexamethylene diisocyanate apparently gave the expected poly-boroureas. Also the reaction was extended to include diethylamino borons and substituted arylisocyanates by the following:

$$\phi B[N(C_2H_5)_2]_2 + 2\ ArNCO \longrightarrow \underset{II}{\phi B[NArCON(C_2H_5)_2]_2} \qquad 2.$$

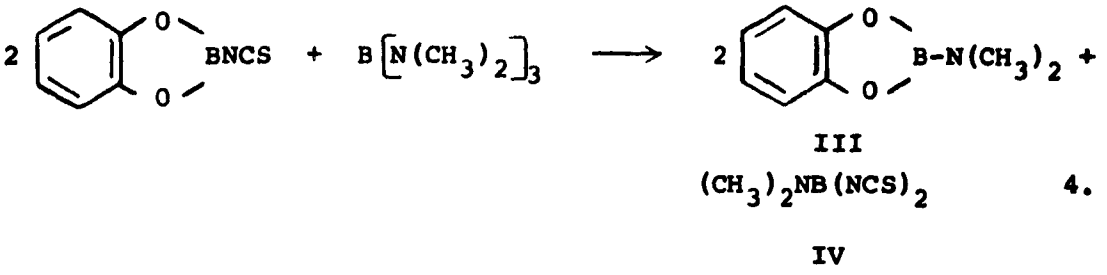
a. - Ar = o-tolyl
b. - Ar = phenyl

If this type of aminoboration could be properly applied to boron isocyanates or boron isothiocyanates, a linear polymer having a B-N backbone could result. This would be an opportunity to arrive at such a linear B-N polymer by a route which does not involve an elimination reaction.

$$\phi B[N(CH_3)_2]_2 + \phi B(NCX)_2 \xrightarrow[\text{give}]{\text{could}} \left[\begin{array}{c} \phi \\ B \\ / \quad \backslash \\ N \quad\quad N \\ | \quad\quad | \\ C{=}X \quad\quad C{=}X \\ | \quad\quad | \\ N \quad\quad N \\ (CH_3)_2 \quad (CH_3)_2 \end{array}\right]_2 \qquad 3.$$

Although the pseudohalogen character of the -NCX group attached to boron is well known to usually favor its displacement rather than permitting it to be added to (e.g. $\rangle B\text{-}NCO + ROH \longrightarrow \rangle B\text{-}OR + (HNCO)$) it was believed that the case at hand could be an exception since cleavage of the $\rangle B\text{-}NCX$ B-N bond in such a displacement would lead only to the formation of another B-N bond (i.e. $\rangle B\text{-}NR_2$).

To first establish a prototype, tris(dimethylamino)boron was allowed to react with o-phenylene isothiocyanato boronate (prepared in good yield by slightly modifying the method of Lappert and Pyszora (5)) in a 1:3 ratio. The desired borourea analog was not obtained and it was subsequently determined that the following disproportionation occurred to give III in 75 percent and IV in 98.5 percent yield.



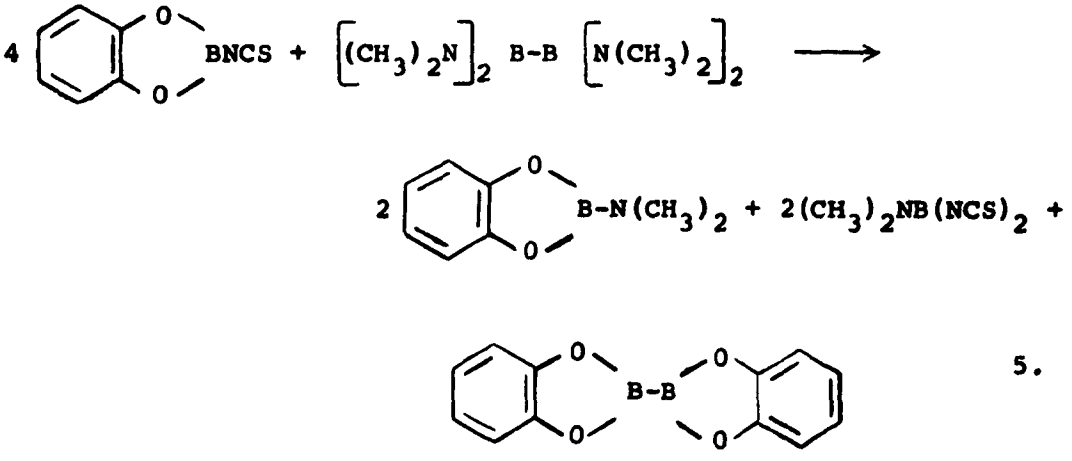
III

$(CH_3)_2NB(NCS)_2$ 4.

IV

In order to identify III it was alternately prepared from o-phenylene chloroboronate and dimethylamine in the presence of triethylamine. Subsequently it was shown that the type of disproportionation in reaction 4 is general whether isothiocyanate or isocyanate groups were involved.

When tetrakis(dimethylamino)diboron was treated with o-phenylene isothiocyanato boronate an interesting reaction occurred which involved not only disproportionation but also cleavage of the boron-boron bond.



5.

The products were isolated in good agreement with this stoichiometry; 2-(1,3,2-benzodioxaborolo)-1,3,2-benzodioxaborole, by comparison of its properties with those previously reported elsewhere (6), was apparently the third product.

When phenylboron diisothiocyanate was heated with an equimolar quantity of bis(dimethylamino)phenylboron and the mixture subsequently distilled, a liquid of constant boiling point was recovered which accounted for more than 90 percent of the weight of starting materials. Its infrared spectrum differed from that of the starting mixture but showed features characteristic of the phenyl, isocyanato and dimethylamino groups. Its mass spectrum, in every detail, was in accord with that of compound V below which was the first example of this type of trisubstituted boron derivative. The use of bis(diethylamino)boron resulted in the formation of VI.

$$\phi B(NCS)_2 + \phi B[N(CH_3)_2]_2 \longrightarrow 2\ \phi B[N(CH_3)_2]NCS \quad 6.$$

V

$$+ \phi B[N(C_2H_5)_2]_2 \longrightarrow 2\ \phi B[N(C_2H_5)_2]NCS \quad 7.$$

VI

The analogous reaction occurred when the corresponding phenylboron diisothiocyanate was replaced by the diisocyanate. Hoping that coordination of the diisothiocyanate with a Lewis base would offset this tendency to disproportionate and permit addition to occur, the pyridine and lutidine adducts were prepared and treated with the bis(dialkylamino)phenyl borons but to no avail.

Since the dialkylaminogroup and the isothiocyanate group were now in the same molecule, attempts were made to induce IV and V to self polymerize. Heating in a sealed system led to the evolution of a considerable quantity of benzene as well as a myriad of other decomposition products. Catalysts such as triethylamine and dibutyltin dioctoate, often useful to promote the formation of polyurethanes from diols and diisocyanates were ineffective.

Final proof that the -NCX groups retained pseudohalogen character in these compounds was gained when diethylamino phenylboronisothiocyanate was treated with ethanol and the boron was completely recovered as diethyl benzeneboronate.

EXPERIMENTAL

Boron-tris-(dimethyl phenylurea) (Ia) Under nitrogen, 3.58 gr. (0.025 mol.) of tris(dimethylamino)boron was added dropwise to a stirred solution of 8.92 gr. (0.075 mol.) of phenyl-isocyanate in pentane during one hour during which time a precipitate formed. After an additional 2.5 hours of stirring, the flask was removed to a dry box where the solution was filtered, the residue washed with pentane and dired under vacuum (yield, 80%). The material could be recrystallized from chloroform but this was found to be unnecessary (m.p. 197°C [a]).

Anal: Calc'd for $C_{27}H_{33}BN_6O_3$: C, 64.80; H, 6.67; B, 2.02; N, 16.80

Found: C, 64.27; H, 7.80; B, 2.00; N, 16.79

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a) Most of these compounds were observed to shrink and undergo other visual changes well below the quoted melting points. These were reproducible and affected by introduction of impurities. Therefore we believe this behavior to be natural for the compounds and not indicative of impurities. This has been verified by other analyses.

I (2.0 gr.) was placed in a 100 ml. flask and 25 ml. of absolute ethanol was added. The flask was heated to 85°C and the volatile materials were distilled away leaving 2.0 g. (100%) of solid identified by infrared, melting point, molecular weight and mixed melting point as phenyldimethylurea. Triethylborate was determined mass spectrometrically in the distillate.

Boron-tris-(dimethyl phenylthiourea) (Ib) A solution of 4.29 gr. (0.03 mol.) of tris(dimethylamino)boron and 12.15 gr. (0.09 mol.) of phenylisothiocyanate in ~75 ml. benzene was refluxed for 41 hours. Some precipitate appeared in the hot solution and on cooling became considerable. This solid (11.0 gr. or 67%) was removed and washed with heptane and dried at 110°C. An additional 1.1 gr. was recovered from the mother liquor. It was found that recrystallization was not necessary to obtain a pure product (m.p. 183-185°C) although this compound is quite soluble in a number of solvents from which it can be recrystallized.

Anal: Calc'd for $C_{27}H_{33}BN_6S_3$: C, 59.10; H, 6.06; B, 1.97; N, 15.30

Found: C, 58.67; H, 7.19; B, 1.70; N, 16.58

5.0 gr. of Ia was refluxed for 1 hour in 50 ml. of absolute ethanol and recovered unchanged. 3.7 gr. of Ia was dissolved in hot dioxane, water added and refluxed for 1.5 hours and concentrated to dryness. The residue was recrystallized from benzene separating the boric acid and giving phenyl dimethyl-thiourea (m.p. 134-136°C and otherwise identified).

Phenylboron-bis-(diethyl o-tolylurea) (IIa) A solution of 4.35 gr. (0.0187 mol.) of bis(diethylamino)phenylboron and 5.0 gr. (0.0376 mol.) of o-tolylisocyanate in 100 ml. pentane was stirred for 56 hours during which time a yellow precipitate formed. This solid was collected, washed with pentane and dried (4.0 g.). Recrystallization from benzene gave a yellow powder melting at 115-116°C.

Anal: Calc'd for $C_{30}H_{39}BN_4O_2$: C, 72.10; H, 7.89; B, 2.21
Found: C, 72.43; H, 7.71; B, 2.14

Phenylboron-bis-(diethyl phenylurea) (IIb) This compound was prepared in essentially the same manner as IIa. It melted at 205-209°C and on hydrolysis gave phenyl diethylurea (m.p. 80-82°C).

Reaction of o-Phenylene Isothiocyanatoboronate and Tris(dimethylamino)boron (reaction 4)

25.0 gr. (0.141 mol.) o-phenylene isothiocyanatoboronate and 50 ml. pentane were stirred at 0°C under nitrogen and to this was added dropwise a solution of 10.1 gr. tris(dimethylamino)boron. A precipitate separated during a 3 hour period and the mixture was allowed to come to room temperature overnight. On cooling in ice the mixture solidified but on warming to 10°C it was filtered. The residue was refluxed in pentane and filtered to give 11.8 g. (98%) of dimethylamino boron-diisothiocyanate (IV) (m.p. 150°C).

Anal: Calc'd for $C_4H_6BN_2S_2$: C, 28.10; H, 3.53; B, 6.33; N, 24.6; S, 37.4
Found: C, 27.79; H, 3.91; B, 6.64; N, 24.3; S, 36.7

Infrared and mass spectral analysis were in complete agreement with the structure of IV.

The filtrate was cooled to 0°C and a precipitate formed which was removed redissolved in pentane, filtered and cooled to give 10.0 gr. of o-phenylene dimethylaminoboronate (III). An additional 7.2 gr. was recovered from the recrystallization liquor of IV (m.p. 59-61°C).

Anal: Calc'd for $C_8H_{10}BNO_2$: C, 59.00; H, 6.19; B, 6.63; N, 8.60

Found: C, 58.77; H, 6.21; B, 6.92; N, 8.81

A compound identical in every respect was prepared from the reaction of equimolar quantities of dimethylamine and o-phenylene chloroboronate in pentane in the presence of triethylamine.

Reaction 4 was repeated using o-phenylene isocyanatoboronate. Due to solubility similarities, separation of the products could not be readily achieved; mass spectral analysis however, was satisfactory to indicate that the same reaction had occurred.

Reaction of o-Phenylene Isothiocyanatoboronate and Tetrakis (dimethylamino)diboron (reaction 5)

o-Phenylene isothiocyanatoboronate (10.0 gr., 0.568 moles) and 50 ml. pentane were placed in 100 ml. flask wrapped with aluminum foil and cooled to < 15°C and to this was added dropwise with stirring, a solution of 2.81 gr. tris(dimethylamino)boron ($\sim$ 5 minutes). A yellow semi-solid had formed but on warming to room temperature it began to turn white and the mixture was allowed to stand overnight. The now hard mass was crushed, agitated in pentane and filtered giving 8.0 gr. of residue. Concentration and cooling of the filtrate gave o-phenylene dimethylaminoboronate.

The residue above was refluxed in benzene and filtered while hot. Pentane was added to the filtrate which was cooled to 0°C

whereupon dimethylaminoboron diisothiocyanate precipitated. The benzene insoluble portion melted at 320-325°C and resembled 2-(1,3,2-benzodioxaborolo)-1,3,2-benzodioxaborole tentatively reported elsewhere (6).

Dimethylamino phenylboronisothiocyanate (V)

To phenylboron diisothiocyanate (11.6 gr., .0568 mol.) and 20 ml. refluxing pentane was added bis(dimethylamino)phenylboron (10.0 g., .0568 mol.) in 20 ml. pentane. The mixture became homogeneous and stirring at room temperature was continued overnight. The pentane was removed and the residue distilled at 110°C/0.5 mm. (18.0, 83%; n_D^{34} = 1.5913).

Anal: Calc'd for $C_9H_{11}BN_2S$: C, 56.80; H, 5.79; B, 5.79; N, 14.75

Found: C, 57.70; H, 6.71; B, 5.63; N, 14.83

The infrared and mass spectrum were in complete agreement with this structure.

Diethylamino phenylboronisothiocyanate (VI)

A solution of 13.2 gr. (.0569 mol.) of bis(diethylamino) phenylboron in 100 ml. benzene was added (10 minutes) to a solution of 11.6 gr. phenylboron diisothiocyanate in 75 ml. benzene and refluxed for 1.5 hours. The benzene was removed and the residue distilled at 155°C/8 mm. to give 24.0 g. (97.1%) of essentially pure diethylamino phenylboronisothiocyanate.

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